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FURTHER INVESTIGATION OF ETCHANTS FOR CHEMICALLY
POLISHING SC-CUT QUARTZ CRYSTALS

RONALD J. BRANDMAYR
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ELECTRONICS TECHNOLOGY & DEVICES LABORATORY



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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) A series of solutions were investigated for chemically polishing SC-cut quartz crystals. Solutions of concentrations (1) $\frac{H_2O}{HF} = \frac{2}{1}$, (2) $\frac{H_2O}{HF} = \frac{3}{2}$, (3) $\frac{H_2O}{HF} = \frac{1}{1}$, (4) $\frac{H_2O}{HF} = \frac{1}{2}$, and (5) $\frac{H_2O}{HF} = \frac{1}{4}$ were prepared. It was determined that some of these solutions can polish SC-cut crystals in less time than solutions previously reported.			

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etching time minimizes the problem of water evaporation from the etching bath. Also, the new solutions are easier to prepare than those employing NH_4F .

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Introduction

The results of experiments aimed at finding a chemical polish for SC-cut quartz crystal plates have been reported previously¹. Some of the solutions evaluated in the previous experiments did not produce a chemically polished surface on either face of the crystal, some produced a polish on one side of the crystal and not the other, and some were able to polish both sides of the crystal. It was shown that an excellent chemical polish could be obtained for both sides of an SC-cut crystal with a solution of NH_4F (40%) : HF (49%) = 4:1. About two hours of etching time was required at 75°C to etch $4f = 15 f_0 f_f^*$. It was also shown that a solution of H_2O : HF (49%) = 4 : 1 could chemically polish SC-cut crystals in two and one half hours at 75°C to the same depth of etch. The purpose of the work described in this report was to investigate additional solutions in the H_2O : HF (49%) series to determine if other such solutions are capable of chemically polishing SC-cut crystals. SC-cut crystals have the potential for providing improved crystal resonators for applications in navigation, communications and identification systems.

Etching Experiments

Five solutions were prepared for this study as follows:

- (1) H_2O : HF = 2 : 1 (2) H_2O : HF = 3 : 2 (3) H_2O : HF = 1 : 1
(4) H_2O : HF = 2 : 3 (5) H_2O : HF = 1 : 2

The HF concentrations were not measured; the 49% specified by the manufacturer was assumed to be correct. The calculated concentrations based on these mixtures are shown in Table 1. The SC-cut crystals were made of natural quartz and had nominal angles of $\theta = 21^\circ 56' \pm 20'$ and $\phi = 34^\circ 10' \pm 5'$. The diameters were 14 mm and the blanks had an initial frequency of 4.060 MHz. The crystals were plano-plano and had $1 \mu\text{m}$ lapped surfaces. Before chemical polishing, the crystals were cleaned thoroughly.

The etching and chemical polishing experiments were carried out at 75°C and, in one instance, at 70°C . Table 1 shows the results of visual inspection of both sides of the crystals etched in the various solutions. A polished surface was obtained on both sides for all solutions except those etched in the H_2O : HF = 1 : 2 solution. Crystals from category were then surface profiled using a Tencor Alpha-step profilometer. An estimate of the surface roughness for each crystal category Alpha-step measurements is shown in Table 2. The surface roughnesses were estimated by calculating the root mean square deviation from an imaginary center line through the Alpha-step profile which was chosen so that the areas under the profile above and below the line were approximately equal, as estimated visually. The values of surface roughness obtained are shown in Table 2.

* f_0 = initial frequency in MHz, f_f = final frequency in MHz

Polished crystals obtained from each of the etching solutions investigated were also examined by scanning electron microscopy. Electron micrographs are shown in figures 1 thru 10 for both sides of each crystal. It can be seen from the electron micrographs that there is a difference in surface roughness between the two sides of the crystals. The difference is more pronounced for the crystals etched in $H_2O : HF = 1 : 1$ based on both the electron micrograph and Alpha-step results. (When tested with an electrometer, the "rough" sides are positive on compression².)

To assure that the surfaces are etched evenly, it is important to remove all contaminants which may be impervious to the etchants³. It should be noted that Ward⁴ has found that the $NH_4F : HF = 4 : 1$ etching solution described previously is more forgiving of surface contamination for SC-cut crystals than the $H_2O : HF = 2 : 1$ solution.

Conclusion

SC-cut quartz plates can be chemically polished on both sides with the designated solutions, at least up to concentrations of $H_2O : HF = 1 : 1$. Starting with $1 \mu m$ lapped surfaces, surface roughnesses of approximately $0.05 \mu m$ and $0.04 \mu m$ can be achieved for the two sides after etching to $\Delta f = 15 f_{off}$. These solutions provide a faster etching rate than those previously reported. For example, a solution of $H_2O : HF = 1 : 1$ at 75° can chemically polish an SC-cut plate to a depth of $\Delta f = 15 f_{off}$ in 42 minutes compared to about two hours for $NH_4F : HF = 4 : 1$ at the same temperature.

Acknowledgements

The authors gratefully acknowledge the contributions of D. Eckart for preparing the SEM micrographs and F. Ivins for providing the Alpha-step profiles.

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1. J.R. Vig, R.J. Brandmayr and R.L. Filler, "Etching Studies on Singly and Doubly Rotated Quartz Plates," Proc. 33rd ASFC, pp. 351 - 358 (1979); Copies available from Electronic Industries Assoc., 2001 Eye Street, N.W. Washington, D.C. 20006.
2. John G. Gualtieri, "A Simple Method for Location of the Mounting Positions for Low Acceleration Sensitivity SC-cut Resonators," Technical Report DELET-TR-81-5, ERADCOM, Fort Monmouth, NJ, Feb 1981.
3. J.R. Vig, J.W. LeBus and R.L. Filler, "Chemically Polished Quartz," Proceedings of the 31st ASFC, pp. 131 - 143, (1977); Copies available from Electronic Industries Assoc., 2001 Eye Street, N.W. Washington, D.C. 20006.
4. R.W. Ward, Colorado Crystal Corp, Private Communication, Jan 1981, also, to be published in the Proceedings of the 35th ASFC, 1981.

TABLE 1

ETCHING RATES

ETCHANT	SIDE 1/SIDE 2	TEMPERATURE °C	TIME OF ETCH (min)	$\frac{\Delta f}{f_o f_f t}$
$\frac{H_2O}{HF} = \frac{2}{1}$ (16% HF)	P/P	75	73	0.22
$\frac{H_2O}{HF} = \frac{3}{2}$ (20% HF)	P/P	75	49	0.32
$\frac{H_2O}{HF} = \frac{1}{1}$ (24% HF)	P/P	75	42	0.36
$\frac{H_2O}{HF} = \frac{2}{3}$ (29% HF)	P/P	75	30	0.51
$\frac{H_2O}{HF} = \frac{1}{2}$ (33% HF)	P/R	70	30	0.68

P = Polished

R = Rough

 f_o = Initial Frequency in MHz f_f = Final Frequency in MHz $\Delta f = (f_f - f_o) 10^3$ in KHz

t = Time in min

TABLE 2

SURFACE ROUGHNESS OF SC-CUT QUARTZ CRYSTALS ($\frac{\Delta f}{f_o f_f} = 15$)

ETCHANT	VERTICAL MAGNIFICATION	ESTIMATED ROUGHNESS (μm)	SIDE
$\frac{\text{H}_2\text{O}}{\text{HF}} = \frac{2}{1}$	100,000X	± 0.05	1
Same	100,000X	± 0.05	2
$\frac{\text{H}_2\text{O}}{\text{HF}} = \frac{3}{2}$	100,000X	± 0.05	1
Same	100,000X	± 0.04	2
$\frac{\text{H}_2\text{O}}{\text{HF}} = \frac{1}{1}$	100,000X	± 0.04	1
Same	100,000X	± 0.04	2
$\frac{\text{H}_2\text{O}}{\text{HF}} = \frac{2}{3}$	100,000X	± 0.05	1
Same	100,000X	± 0.04	2
$\frac{\text{H}_2\text{O}}{\text{HF}} = \frac{1}{2}$	100,000X	± 0.04	1
Same	100,000X	± 0.09	2

* Measured with a TENCOR INSTRUMENTS Alpha-Step profile meter
Horizontal Magnification: 50X
Distance measured: 6mm

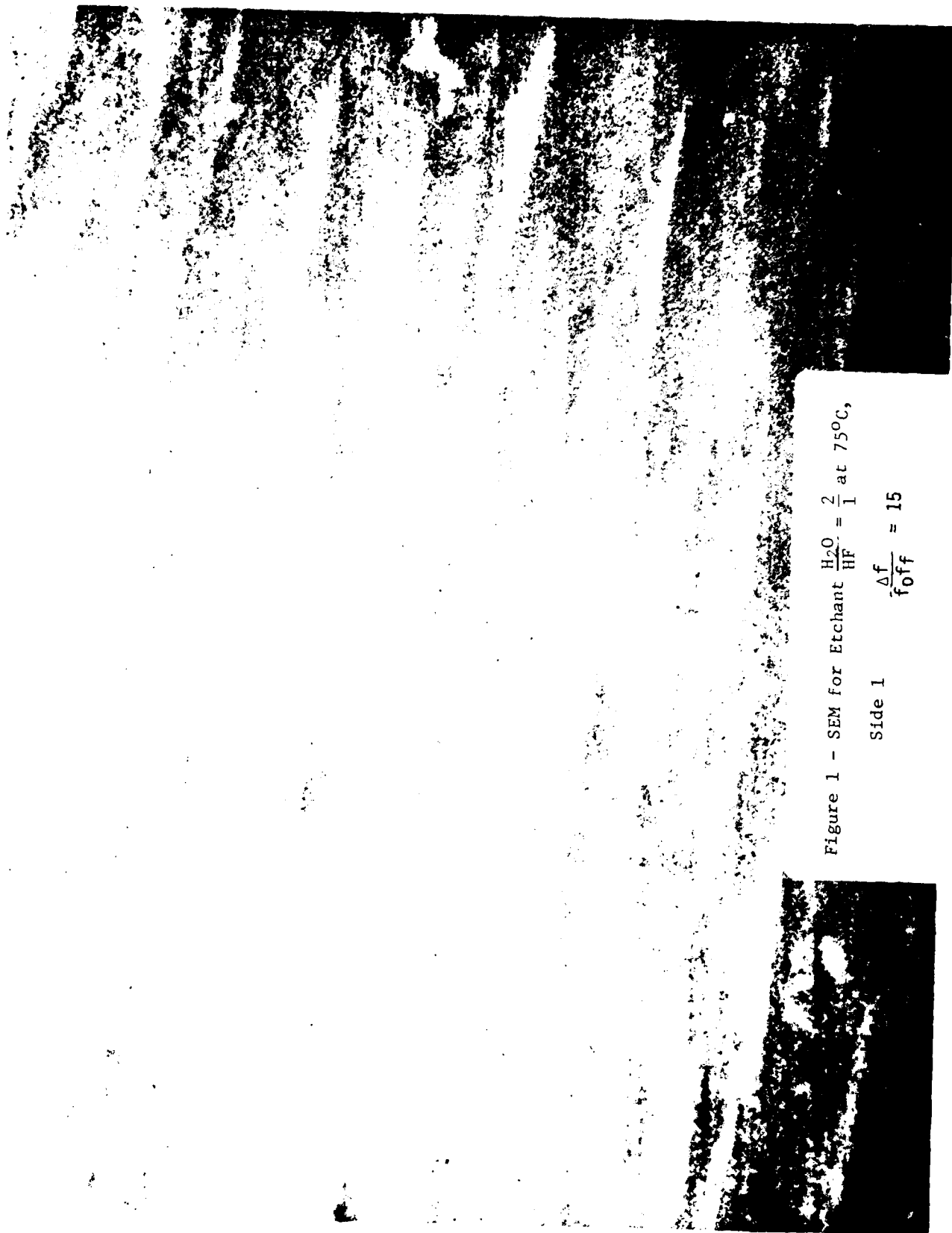


Figure 1 - SEM for Etchant $\frac{\text{H}_2\text{O}}{\text{HF}} = \frac{2}{1}$ at 75°C,

Side 1

$$\frac{\Delta f}{f_{\text{off}}} = 15$$



Figure 2 - SEM for Etchant $\frac{\text{H}_2\text{O}}{\text{HF}} = \frac{2}{1}$ at 75°C,

Side 2 $\frac{\Delta f}{f_o f_f} = 15$

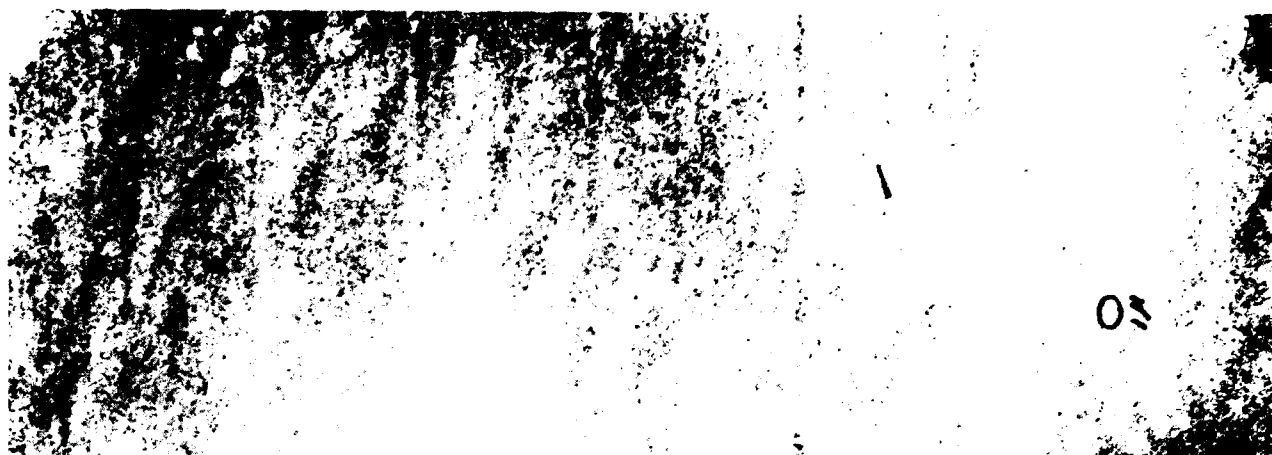


Figure 3 - SEM for Etchant $\frac{\text{H}_2\text{O}}{\text{HF}} = \frac{3}{2}$ at 75°C,

Side 1 $\frac{\Delta f}{f_{of}} = 15$

0/4

Figure 4 - SEM for Etchant $\frac{\text{H}_2\text{O}}{\text{HF}} = \frac{3}{2}$ at 75°C,

Side 2 $\frac{\Delta f}{f_{off}} = 15$

0.1

Figure 5 - SEM for Etchant $\frac{\text{H}_2\text{O}}{\text{HF}} = \frac{1}{1}$ at 75°C,

Side 1 $\frac{\Delta f}{f_0 f_f} = 15$



Figure 6 - SEM for Etchant $\frac{\text{H}_2\text{O}}{\text{HF}} = \frac{1}{1}$ at 75°C ,

Side 2 $\frac{\Delta f}{f_{of}} = 15$

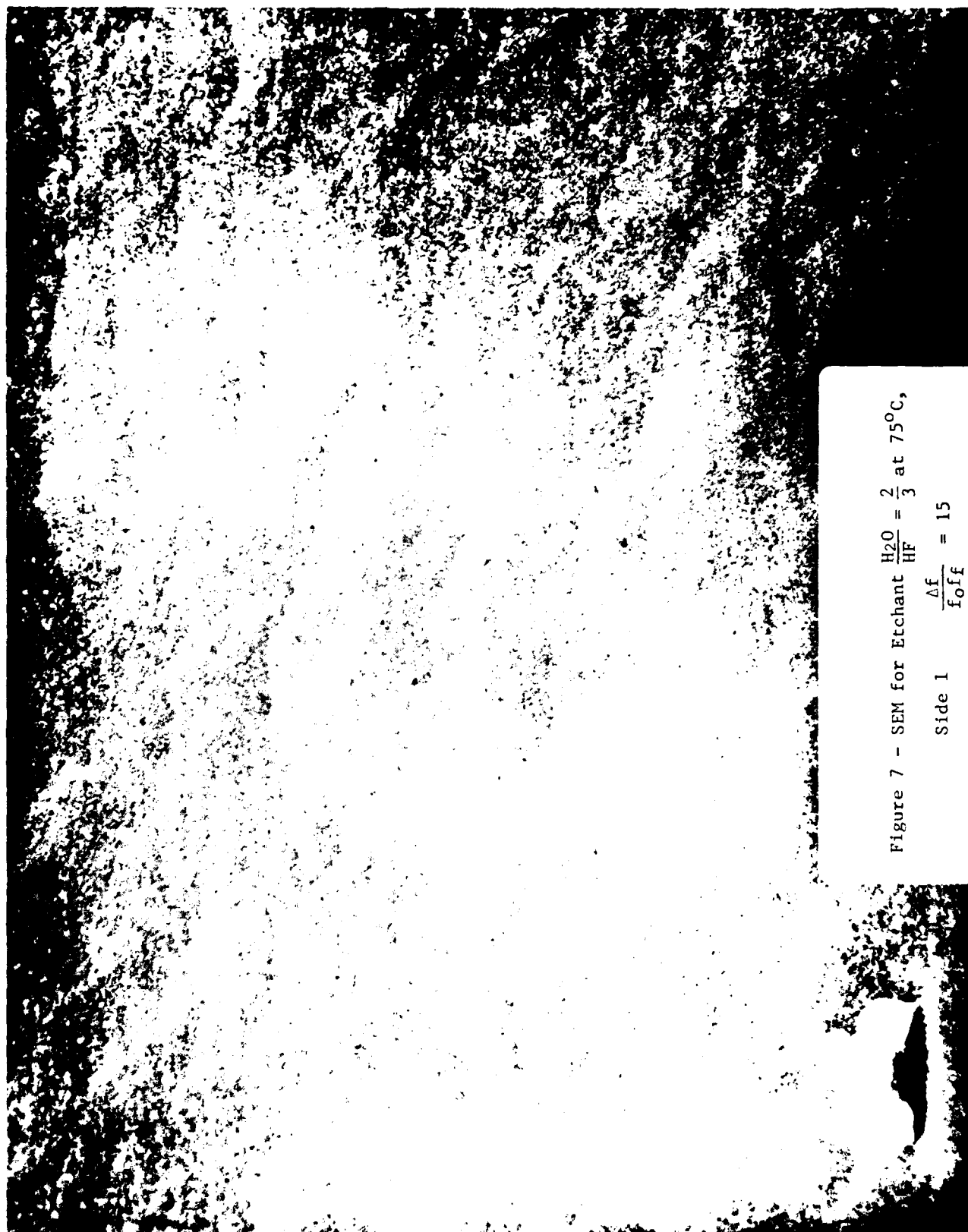


Figure 7 - SEM for Etchant $\frac{\text{H}_2\text{O}}{\text{HF}} = \frac{2}{3}$ at 75°C ,

Side 1 $\frac{\Delta f}{f_{\text{off}}} = 15$



Figure 8 - SEM for Etchant $\frac{\text{H}_2\text{O}}{\text{HF}} = \frac{2}{3}$ at 75°C ,

Side 2 $\frac{\Delta f}{f_{\text{off}}} = 15$

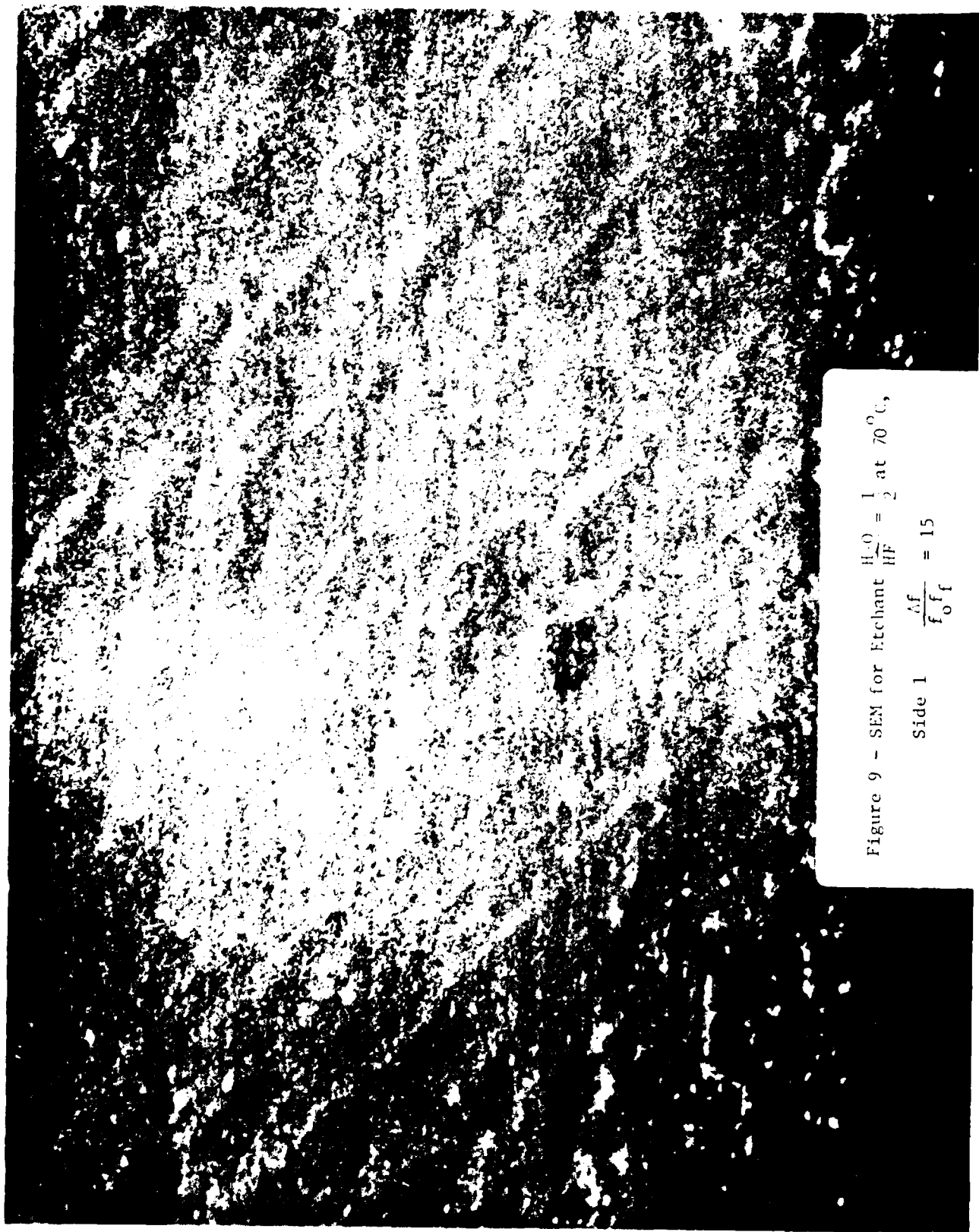


Figure 9 - SEM for Etchant $\frac{H_2O}{HF} = \frac{1}{2}$ at $70^\circ C$,

Side 1 $\frac{\Delta f}{f_o f_i} = 15$

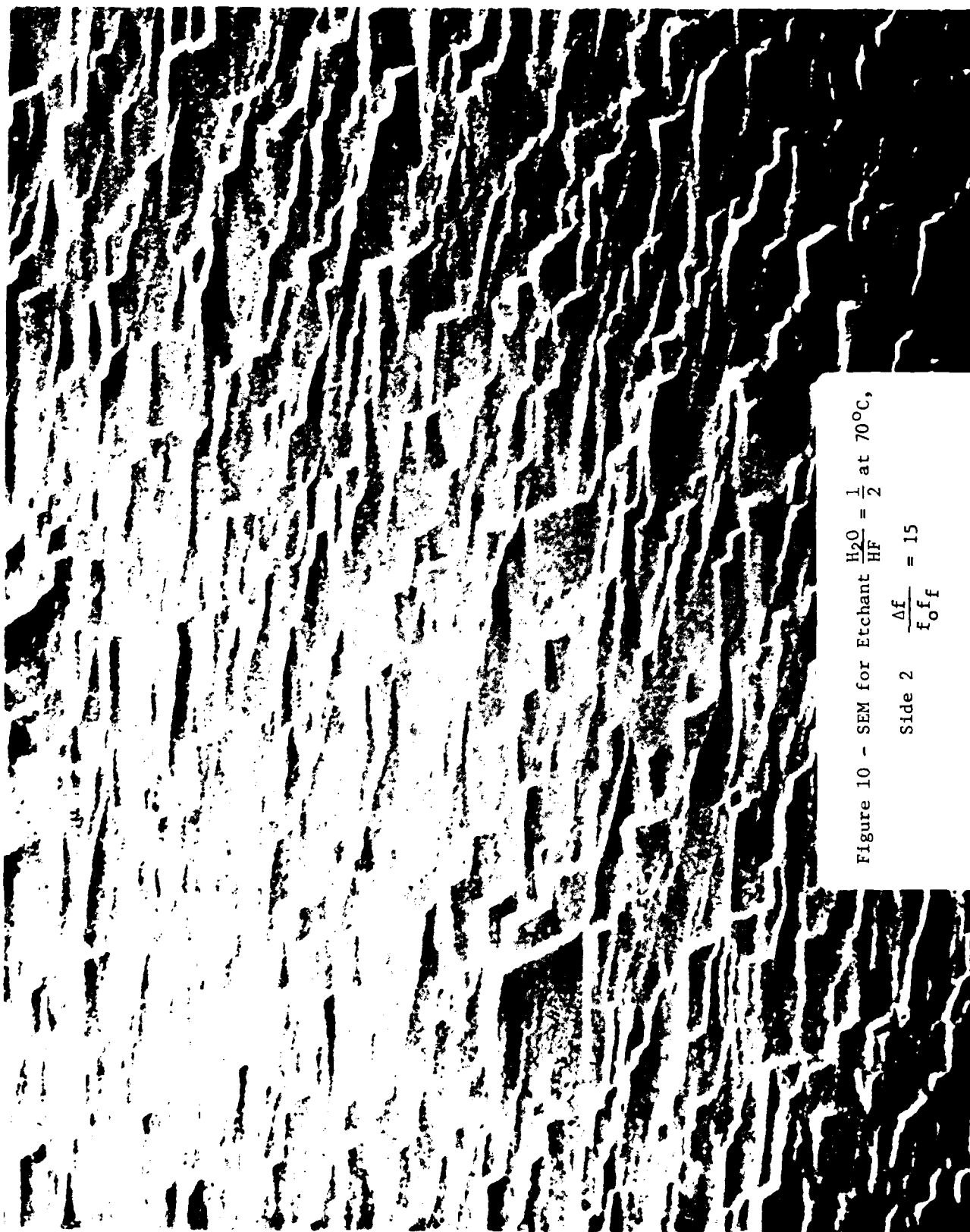


Figure 10 - SEM for Etchant $\frac{\text{H}_2\text{O}}{\text{HF}} = \frac{1}{2}$ at 70°C ,

Side 2 $\frac{\Delta f}{f_o f_f} = 15$